

WHAT IS CLAIMED IS:

1. A method of reducing DC leakage comprising milling niobium chips at elevated temperatures and in the presence of at least one liquid solvent to form niobium powder and forming said niobium powder into a capacitor anode.
- 5 2. The method of claim 1, wherein said elevated temperature is at least 300° F.
3. The method of claim 1, wherein said elevated temperature is at a temperature of from about 300° F to about 420° F.
4. The method of claim 1, wherein said elevated temperature is from about 330° F to about 380° F.
- 10 5. The method of claim 1, wherein said liquid solvent comprises tetraethylene glycol, polyethylene glycol, or mixtures thereof.
6. The method of claim 1, wherein said liquid solvent comprises at least one glycol.
7. The method of claim 1, wherein said liquid solvent is present in an amount of from about 1/2 gallons to about 2 gallons of liquid solvent per 3 to 5 pounds of niobium powder.
- 15 8. The method of claim 1, wherein said milling is ball milling.
9. The method of claim 1, wherein said milling is accomplished in an attritor ball mill.
10. The method of claim 1, wherein said milling occurs for a time of from about 30 minutes to about 10 hours.
11. The method of claim 1, further subjecting the niobium powder to deoxidization after said milling and then continuing said milling of said niobium powder at elevated temperatures and in the presence of at least one liquid solvent.
- 20 12. The method of claim 11, wherein the step of subjecting the niobium powder to deoxidization is repeated one or more times during the milling of the niobium powder.
13. The method of claim 11, wherein said deoxidization comprises an inorganic deoxidization.
- 25 14. The method of claim 11, wherein said deoxidization is a magnesium deoxidization.
15. The method of claim 11, wherein said milling is accomplished by an attritor mill.
16. A method of reducing the DC leakage in a valve metal comprising milling the valve metal at elevated temperatures and in the presence of at least one liquid solvent to form valve metal powder and forming said valve metal powder into a capacitor anode.
- 30 17. The method of claim 16, wherein said elevated temperature is at least 300° F.
18. The method of claim 16, wherein said elevated temperature is a temperature of from about 300° F to about 420° F.
19. The method of claim 16, wherein said elevated temperature is from about 330° F to about 380° F.
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20. The method of claim 16, wherein said liquid solvent comprises tetraethylene glycol, polyethylene glycol, or mixtures thereof.

21. The method of claim 16, wherein said liquid solvent comprises at least one glycol.

22. The method of claim 1, wherein said niobium powder is flaked.

5 23. The method of claim 11, wherein said niobium powder is flaked.

24. A method of making niobium powder comprising milling niobium chips at elevated temperatures and in the presence of at least one liquid solvent to form niobium powder.

10 25. A method of reducing the milling time to form a high surface area metal powder comprising milling metal chips at elevated temperatures and in the presence of at least one liquid solvent to form metal powder.

26. A method of making a metal powder comprising milling metal chips at elevated temperatures and in the presence of at least one liquid solvent to form metal powder.

15 27. A method to reduce the amount of contaminants in a niobium powder comprising milling said niobium powder at elevated temperatures and in the presence of at least one solvent to form a niobium powder having reduced contaminants.

28. A niobium powder having a carbon content of from about 40 ppm to about 200 ppm and a iron, nickel, and chromium content of from about 5 ppm to about 200 ppm.

29. The niobium powder of claim 28, wherein said carbon amount is from about 50 to about 150 ppm.

20 30. A niobium powder having a carbon amount of from about 40 to about 200 ppm when the BET surface area is about $1.0 \text{ m}^2/\text{g}$ and a carbon amount of less than 250 ppm when the BET surface area of the niobium powder is from about 2 to about $4.5 \text{ m}^2/\text{g}$ and the combined amount of Fe/Ni/Cr is less than 100 ppm when the BET surface area of the niobium powder is about $1.0 \text{ m}^2/\text{g}$ and less than about 400 ppm when the BET surface area of the niobium powder is from about $2.0 \text{ m}^2/\text{g}$ to
25 about $4.5 \text{ m}^2/\text{g}$.

31. A capacitor anode comprising the niobium powder of claim 28.

32. A capacitor anode comprising the niobium powder of claim 29.

33. A capacitor anode comprising the niobium powder of claim 30.

34. The method of claim 1, wherein said liquid solvent is at least one fluorinated fluid.

30 35. A method of making a flaked metal comprising wet-milling a metal powder in the presence of at least one fluorinated fluid.

36. The method of claim 35, wherein said fluorinated compound is a perfluorinated compound.

- 37. The method of claim 35, wherein said metal is a valve metal.
- 38. The method of claim 35, wherein said metal is niobium.
- 39. The method of claim 35, wherein said metal is tantalum.
- 5 40. The method of claim 16, wherein said metal is a valve metal.
- 41. The method of claim 40, wherein said valve metal is tantalum.
- 42. A method of making valve metal powder comprising milling valve metal chips at
elevated temperatures and in the presence of at least one liquid solvent to form valve metal powder and
then subjecting the valve metal powder to deoxidization and then continuing said milling of said valve
10 metal powder at elevated temperatures and in the presence of at least one liquid solvent.
- 43. The method of claim 16, wherein said valve metal is tantalum.